#### SOLVAY CHEMICAL, INC. GREEN RIVER, WYOMING

# CALCINERS 1 AND 2 COMMON STACK HYDROGEN FLUORIDE EMISSIONS REPORT

Report submitted on May 30, 2013 to:

Mr. Tim Brown
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Solvay Chemicals, Inc.
20 Miles West of Green River
Green River, WY 82935

Report Prepared by:



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We certify that we have examined the information submitted in this report and believe the results presented are true, accurate, and complete.

Daniel Klassen

President

Justin Russell Quality Control

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#### 1.0 PROJECT OVERVIEW

Solvay Chemicals, Inc. Plant (Solvay) contracted Optimal Air Testing Services, Inc. (Optimal) to complete air emissions testing. Emissions of hydrogen fluoride from the Calciners 1 & 2 common stack were measured for compliance purposes. The Calciner 1 & 2 common stack is also referred to as "CA-1 & 2" and Calciner Stack "CA-A & B" common stack. Test program results are summarized below.

Testing was coordinated by Tim Brown and Ouisha Toenyes, Solvay and Mr. Dan Klassen, Optimal. Testing followed procedures set forth in the <u>Code of Federal Regulations</u>, Title 40 (40 CFR), Part 51, Appendix M and Part 60, Appendix A.

The scope of work was to complete three runs simultaneously measuring flow rate and hydrogen fluoride emissions. Parameters measured to calculate flow rate include velocity, temperature and concentrations of moisture, oxygen  $(O_2)$  and carbon dioxide  $(CO_2)$ . Average results for the three run, summarized below in Table 1, were based on the laboratory limit as hydrogen fluoride was not detected in the samples. Test parameters for each run are detailed in Section 2, Table 3.

Field data recorded on-site, attached in Appendix A, was entered into spreadsheets and used to calculate results, as shown in Appendix B. Equipment calibrations are attached in Appendix C. Example equations are presented in Appendix D and laboratory data is included in Appendix E.

Table 1 CA - 1 & 2 Summary of Hydrogen Fluoride Test Results, Solvay, April 19, 2012

Parameter / Pollutant	EPA Method	Units	Measured Results (average)
Stack Gas Temperature	2	°F	401
Stack Gas Moisture	4	%	23.47
Oxygen (O <sub>2</sub> ) Carbon Dioxide (CO <sub>2</sub> )	3A	%	13.30 9.30
Flow Rate	1-4	acfm dscfm	388,443 144,077
HF Concentration in Gas	26A	gr/dscf lb/hr lb/ton of coal	<0.00004 <0.049 <0.0044



#### 2.0 SUMMARY OF RESULTS

Table 2 CA - 1 & 2 Hydrogen Fluoride Test Parameters and Results, Solvay, April 19, 2013

Table 2 CA - 1 & 2 Hydrogen FI	Start Date/Time		4/19/13 12:25	4/19/13 14:08	
	Start Date/Time Stop Time	12:11	13:58	15:30	
Coal Consumption	Units	Run 1	Run 2	Run 3	
AQD #17 "A" Calciner	TPH	5.6	5.6	5.6	-
AQD #17 "B" Calciner	TPH	5.6	5.6	5.6	
Combined Coal Consumption	TPH	11.2	11.2	11.2	
Test Parameters	<u>Units</u>	Run 1	Run 2	Run 3	Avg.
P <sub>bar</sub> (Barometric Pressure, absolute)	In. Hg	23.70	23.70	23.70	
Y (Dry Gas Meter Calibration Factor)	unitless	0.992	0.992	0.992	
C <sub>p</sub> (Pitot tube Coefficient)	unitless	0.84	0.84	0.84	
D <sub>n</sub> (Diameter of Nozzle)	Inches	0.304	0.304	0.304	
θ (Total Sampling Time of Test)	Minutes	80	80	80	
△H (Orifice Pressure Drop)	In. H2O	1.87	1.94	2.04	
V <sub>m</sub> (Dry Gas Sampled - as measured)	ft <sup>3</sup> (dry)	61.435	63.796	66.612	
T <sub>m</sub> (Gas Meter Temperature, avg.)	Degr. F	83	87	87	
V <sub>lc</sub> (Condensate and silica gel)	ml or g	306.5	316.6	330.8	
<b>Location/Process Parameters</b>					
A <sub>s</sub> (Cross-sectional Area of Stack)	$\mathrm{ft}^2$	113.1	113.1	113.1	113.1
P <sub>g</sub> (Static Pressure of Stack Gas)	In. H2O	-0.60	-0.61	-0.60	-0.60
T <sub>s</sub> (Temperature of Stack Gas)	Deg. F	401	400	402	401
$\sqrt{\Delta p}$ (Sq. root of velocity head of gas)	√ In. H2O	0.6902	0.6829	0.6940	0.6890
CO <sub>2</sub> (Carbon Dioxide)	%	9.4	9.4	9.0	9.3
O <sub>2</sub> (Oxygen)	%	13.0	13.4	13.4	13.3
<u>Calculations</u>					
V <sub>mstd</sub> (Gas Sampled, standard (std) cond.)	$\mathrm{ft}^3$	47.17	48.68	50.78	48.88
V <sub>wstd</sub> (Water Vapor in Gas Sampled, std)	$\mathrm{ft}^3$	14.45	14.93	15.60	14.99
B <sub>ws</sub> (Water Vapor in Gas, by Vol.)	%	23.45	23.47	23.50	23.47
M <sub>d</sub> (Molecular Weight of Dry Stack Gas)	lb/lb-mole	30.02	30.04	29.98	30.01
M <sub>s</sub> (Molecular Weight of Wet Stack Gas)	lb/lb-mole	27.20	27.21	27.16	27.19
P <sub>s</sub> (Pressure of Stack Gas, Absolute)	In. Hg	23.66	23.66	23.66	23.66
Iso (Percent of Isokinetic Sampling)	%	91.7	95.6	98.2	95.2
Flow Results					
V <sub>s</sub> (Average Stack Gas Velocity)	ft/m (fpm)	3,440	3,400	3,464	3,435
Q <sub>a</sub> (Actual Volumetric Flow Rate)	ft <sup>3</sup> /m (cfm)	389,040	384,540	391,750	388,443
Q <sub>std</sub> (Dry Volumetric Flow Rate, std.)	ft <sup>3</sup> /m (dscfm)	144,340	142,850	145,040	144,077
HF Results		-	_	-	
Mass of HF Collected	mg/sample	< 0.123	< 0.126	< 0.128	
HF Concentrations in gas - std.	$mg/m^3$ gas	< 0.092	< 0.094	< 0.096	
HF Concentrations in gas - std.	gr/dscf	< 0.00004	< 0.00004	< 0.00004	
HF Concentrations in gas	ppm	< 0.111	< 0.113	< 0.115	< 0.113
HF Emission Rate (Coal Fd = 9780)	lb/mmBtu	<1.5E-04	<1.6E-04	<1.5E-04	<1.5E-04
HF Emission Rate	lb/hr	< 0.050	< 0.049	< 0.048	< 0.049
HF Emission Rate	lb/ton of coal	< 0.0044	< 0.0044	< 0.0043	< 0.0044



#### 3.0 PROCESS DESCRIPTION

Solvay Chemicals, Inc., located near Green River, Wyoming, is a trona mine and refinery with corporate offices in Houston, Texas.

The primary raw material for the Green River facility is sodium sesquicarbonate, commonly referred to as trona. The trona is mined at the plant site from an ore bed located 1,500 feet below the surface. The trona is hoisted to the surface before refining into soda ash and other sodium-based products.

The trona that is fed to the soda ash calciners is heated, resulting in thermal calcinations of the sodium sesquicarbonate forming a crude soda ash. The crude soda ash is dissolved in water and the insolubles are separated from the solution by settling and filtration. The insolubles are disposed of in the mine void. The high-purity saturated solution of sodium carbonate is then fed to crystallizers where a large amount of water is removed and a slurry of sodium carbonate monohydrate crystals is formed. This slurry is then further dewatered and washed by a series of cyclones and centrifuges. The resulting monohydrate crystals are fed through dryers forming a high quality soda ash, which then is ready for storage and shipment.



#### 4.0 SAMPLING AND ANALYTICAL PROCEDURES

Optimal Air Testing Services, Inc. collected source data and samples of gas from the CA - 1 & 2 exhaust stack under the roof (inside building). Sampling activities were performed in accordance with standard EPA sampling methods listed in 40CFR51, Appendix M and 40CFR60, Appendix A. The parameters and pollutants measured during the sampling program are listed below with brief descriptions of the sampling methods.

• Traverse to measure velocity head and temperature. Method 1 was used to calculate the points (location) in the stack for sampling. Method 2 procedures were followed to measure stack gas velocity and temperature in conjunction with particulate sampling.

The state of the s	
CA - 1 & 2 Stack Configuration	Vertical – circular
Test Location	Stack
Measured Inside Stack Diameter	144 inches
Port Length	Port A – 9.0 inches Ports B, C and D – 9.5 inches
Distance from ports upstream to disturbance	~66 feet (~5.5 diameters)
Distance from ports downstream to disturbance	~80 feet (~6.7 diameters)
No. of Ports	4
Velocity/temp./Particulate traverse points	20 (5 per port)
Point #1	$3^{3}/_{4}$ (probe mark at $13^{1}/_{4}$ )
Point #2	$11^{13}/_{16}$ (probe mark at $21^{5}/_{16}$ )
Point #3	21 (probe mark at $30^{-1}/_{2}$ )
Point #3	$32^{9}/_{16}$ (probe mark at $42^{1}/_{16}$ )
Point #4	$49^{1}/_{4}$ (probe mark at $58^{3}/_{4}$ )

Table 3 CA - 1 & 2 Stack Dimensions and Traverse Points, Solvay

- Oxygen, Carbon Dioxide and Molecular Weight. Stack gas molecular weight was calculated from oxygen and carbon dioxide concentrations measured from a gas sample collected during each run. O<sub>2</sub> and CO<sub>2</sub> concentrations were measured in accordance with 40 CFR 60 Method 3A (instrument analyzers) in conjunction with the NOx sampling, as described below.
- Moisture. Stack moisture measurements was calculated from the volume of moisture that
  condensed out of a measured volume of exhaust gas collected following 40 CFR 60 Method 4.
  Moisture was measured in conjunction with particulate sampling.
- Flow Rate. Stack gas flow rate was calculated for each run from the velocity, molecular weight, and moisture content determined via Methods 1 through 4.
- **Hydrogen Halogens and Halides.** Samples were collected isokinetically in accordance with 40 CFR 60, Method 26A and analyzed for HCl.



#### 4.1 Temperature, Velocity, Moisture and Flow Rate

Method 1-4 procedures were used to select sampling locations, operate the sampling train, record data, and calculate flow rate in conjunction with the isokinetic hydrogen fluoride sampling trains.

Stack samples were collected from five points in each of the four ports, for a total of twenty points. The sampling point locations for the stack are listed in Table 3.

Sampling equipment was calibrated at the Optimal's laboratory prior to job mobilization. Equipment calibrations are included in Appendix C.

The velocity sampling apparatus consisted of S-type stainless steel pitots used to measure velocity head (pressure) and a thermocouple to measure temperature. Tubing connected the pitot tubes to an inclined manometer. The velocity apparatus was leak checked before and after each run.

Moisture concentration was determined by drawing a measured volume of the stack gas through chilled impingers. Moisture condensed in the impingers as the gas was cooled below 68°F. The total weight gain of the impingers and the volume of gas were measured to calculate the moisture concentration in the stack gas. The sampling train was checked for leaks before and after each run.

#### 4.2 Hydrogen Halides and Halogens

EPA Methods 26A was used to determine hydrogen fluoride (HF) concentrations at the test location. A sample of the gas stream was withdrawn from the source isokinetically. The HF was collected in a solution of 0.1 N sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Hydrogen fluoride analysis was by ion chromatography.

#### **Pretest Preparation**

All glassware was cleaned with detergent and rinsed with tap water. Then all glassware was cleaned with  $0.1~N~H_2SO_4$ , distilled water and acetone prior to shipment to the test location. The meter, thermometers, and pitot tube were calibrated prior to shipment. Sampling nozzles were calibrated on site.

#### **Apparatus**

The probe nozzle was made of borosilicate glass of buttonhook design with a taper angle of less than 30°. The probe was constructed of borosilicate glass and equipped with a heater capable of maintaining a constant temperature above  $248^{\circ}F \pm 25^{\circ}F$  for the test duration, to prevent moisture condensation in the probe. The probe was equipped with a pitot tube for constant monitoring of the stack gas velocity and a thermocouple accurate to within  $\pm 2^{\circ}F$  to measure the stack gas temperature. The filter holder was made of borosilicate glass with a Teflon support and a Teflon coated viton gasket to provide a positive seal. A filter heating system capable of maintaining a temperature above  $248^{\circ}F \pm 25^{\circ}F$  and a thermocouple accurate to within  $\pm 2^{\circ}F$  were utilized. The metering system included: a vacuum gauge, leak free pump, thermometers accurate to within  $\pm 5.4^{\circ}F$  and a dry gas meter accurate to within 2 percent. Differential pressure gauges were used, one to measure stack gas velocity and the other for orifice differential pressure readings. The first and second impingers contained 100-ml of 0.1 N H<sub>2</sub>SO<sub>4</sub>, the third was empty, and the fourth impinger contained a tare weighted quantity of silica gel.



#### Reagents

Teflon filters with 99.95% efficiency, <0.05% penetration, on 0.3-micron dioctyl phthalate smoke particles were used. Acetone, distilled water, and 0.1 N H<sub>2</sub>SO<sub>4</sub> of reagent grade were used.

#### Sampling

The sample points were selected according to procedures outlined in EPA Method 1. The stack pressure, temperature, and velocity heads were determined using EPA Method 2. The nozzle size was selected for isokinetic sampling based upon the velocity head range.

The sample train was assembled using ball joint style glassware with Teflon coated o-rings on the joints to ensure a leak free seal. The impingers were placed in an ice bath for the duration of the test.

The entire sampling train was leak checked and had <0.02-cfm leakage prior to any sampling. The probe was placed in the stack and the system allowed to heat up. The sampling commenced when the probe and filter reached sampling temperature. Sampling time was increased to 80 minutes in order to reduce the detection limit.

After the test, the sample train was leak checked. The leakage was less than the maximum allowable amount (0.02 cfm or 4% of the average sampling rate) and test results were considered allowable.

#### Sample Recovery

A 100-ml of acetone, a 100-ml sample of the  $0.1 \text{ N H}_2\text{SO}_4$ , and a 200-ml sample of distilled water were taken as reagent blanks. The tared drying column (silica gel) was removed and weighed, the weight gain of the desiccant recorded and used in the moisture calculations. The impinger catch was weighed on top loader balance to determine the weight to the nearest 0.5 gram, which was used in the moisture calculation. The  $0.1 \text{ N H}_2\text{SO}_4$  impinger contents were recovered and placed into a 500-ml glass sample jar with a Teflon lined lid. The connecting glassware and the two acid impingers were rinsed with distilled water and combined with the acid impinger contents. The liquid levels were marked for transportation to the laboratory.

#### Analysis

The hydrogen fluoride was analyzed by ion chromatography.



APPENDIX A
FIELD DATA

# Source Testing Field Data Sheet for Hydrogen Fluoride

Jo

Page

Run

Methods 1-5/26A

Client:Solvav Chemical	Motor ID 11	Date: & 11 1.11.1	Imninger Weight	icht		Immino	
Chemical vay Chemical	Meter ID	Date: 19 19/19	mpinger we	ıgııı		unpinger	CI
Plant: Green River	Meter Y 0.992	Ambient Temp. F: 80	Pretest (g) Post test (g) Total (g) Vol. (ml) Style* Contents	g) Total (g)	Vol. (ml)	Style* (	ontents
Test Location: Calciner	Meter ΔH@ 7.00%	Bar. Pressure in. Hg:	5-00+ 8'94C	400-9013 1345 ~100	2012	WGS*	MGS* 0.1N H <sub>2</sub> SO <sub>4</sub>
Unit: CA 1 & 2 (aka CA- A & B)	Pitot ID p - 4 - 2	Static Press. in. H <sub>2</sub> O +QO, 53	27867 9446	626	001 ~ 626		GS* 0.1N H <sub>2</sub> SO <sub>4</sub>
Project No.: 1301A	Pitot Cp © 344	Assumed % Moist	3658,4 688,0	29.6	)	WGS*	
Meter Operator: 7 Aussell	Probe Liner $\mathcal{L}\{ass\}$	K Factor 3,3	4939.2 9637	245	245 ~3004	WGS*	MGS* Silica Gel
Assistant: () Klussen	Nozzle ID 6, 1454	Duct Dimensions: 144 inches	5	3065	7		
Nozzle Calibration	Nozzle Size 0 304	Port Length: 28 inches 95	Filter NA (Teflow				
1 <sup>st</sup> Dia. 0,303	Sample Time &	Pitot Passes Leak Checks:	Inside Boilding		CO2	$CO_2$ $CO_2+O_2$	O <sub>2</sub>
2nd Dia. 0.304	% CO2 9.4	Pretest Posttest Diluent Analysis	Diluent Analysis	1st Orsat	5.6	22,4	(3,0
3rd Dia. 6.304	% O <sub>2</sub> 13,0	1st Point all the way In Out	by CEMS $\Box$	2 <sup>nd</sup> Orsat			

Leak Rates/Notes	Pre-test 4 in Hg G. co. offm.	Post-test 9 in Hg 0.003cfm					8.0	à										Kfurter 3.8				Ontimal Air Testing	
Pump	Vacuum	(in. Hg)	4.5	5.5	5.51	5.4	Sit of	5,0	2,0	2,5	2.5	7.0	0.0	6.0	6.0	0.0	5.5	7.0	7.0	2.5	2.5	5.0	
	023		++	326	360	364	363	361	36	260	26,	361	363	255	358	259	Sel	363	360	3.59	300	259	
		Probe	36	395	R	979	369	358	38	R	2,70	360	346	395	287	271	30	365	266	256	250	250	4
iture, °F	eter	Outlet .	79	29	79	79	79	79	41	85	208	18	83	27	33	176	25	84	18	84	35	48	
Temperature,	Gas Meter	Inlet	80	76	75	73	79	21	83		200	87	80	84	89	89	06	80	05	88		83	-
	Impinger	Outlet	00	63	58	59	100	63	63	29	67	e	67	3	64	62	(01	60	28	57	53	59	
		Stack	404	409	117	412	410	461	405	401	403	400	393	393	392	341	341	403	407	107	401	395	
Gas Meter	ft <sup>3</sup>	688,765	691.83	194.97	693, 11	701.07	703.846	706.87	710,19	713.59	716.95	730,024	723,69	726.13	729, 19	732.13	734,843	738,05	741.23	744.51	747.91	750.200	
Orifice	Setting		3.0	0.0	3.0	1.7	1.1	2,0	7.7	4.8	2.7	4	5.7	00	. s	1.7	1	2.0	3,0	2.2	2,0	0/	
Velocity	Head	0 (Δp, in. H <sub>2</sub> O)	0.51	6.52	0.0	0.44	0.35	0.50	0.65	00.0	B.50	O.4(	0.47	6.45	0,40	ナナ・ロ	0.37	6.63	6.53	0.57	0.52	0.37	
Time	(minutes)	10 HH 01	1	F	C	16	1104 20	106 24	24	32	36	0h 0/2)	My 800	48	50	56	149 60	15164	63	72	20	6.0	
Traverse	Point	(Port-Pt)	05	-4	-3	-2	-1 1	C -51	-4	-3	-2	-1	B -5 1129	4-	-3	-2	1	4 -5	-4	-3	-2	-1	Avg /Total

Source Testing Field Data Sheet for Hydrogen Fluoride

Methods 1-5/26A

Run

0.1N H<sub>2</sub>SO<sub>4</sub> GS\* 0.1N H<sub>2</sub>SO<sub>4</sub> MGS\* Silica Gel ó Vol. (ml) |Style\* |Contents Impinger CO, CO,+O, MGS\* WGS\* 3 2 100 Pretest (g) Post test (g) Total (g) 3/6/6 224.2 13.8 72,0 e è Impinger Weight 975.6 662.8 701.3 Filter Toflow 6293 656,2 VNOPP Duct Dimensions: 144 inches Bar. Pressure in. Hg: 33.7 Static Press. in. H<sub>2</sub>O +  $\bigcirc$   $\bigcirc$   $\bigcirc$ Port Length: 3.5 inches 9.5 Pitot Passes Leak Checks: Ambient Temp. °F: 82 Pretest W Pc 1st Point all the way I Assumed % Moist Date: 4/14/13 K Factor Nozzle Size 6,304 Meter AH(a) 2,008 Pitot ID p - 4 - 2 Probe Liner Cluss Sample Time 80 Nozzle ID Glass Pitot Cp 0,84 Meter Y 0,942 45 13.4 Meter ID % CO<sub>2</sub> % 02 Unit: CA 1 & 2 (aka CA- A & B) Meter Operator: J Russey See Ranl Nozzle Calibration Assistant: D Klass Client:Solvay Chemical Test Location: Calciner Project No.: 1301A Plant: Green River 2<sup>nd</sup> Dia. 3<sup>rd</sup> Dia.

	nalysis	1st Orsat	414	14 22.8	(3,4	
DOut 6	by CEMS 🗇	2 Orsat				

es/Notes	in Hg 0.005 cfm.	in Hg 0,003 cfm						7														Optimal Air Testing	
Leak Rates/Notes	Pre-test	Post-test 9,5 ir	1 1					K factor 4.			254 254											Optimal 4	
Pump	Vacuum	(in. Hg)	5.0	0.0	6.0	2,5	4,5	52	12	5.5	55	0.5	0.3	7.0	7.5	7.5	6.0	7.0	7.0	7.0	2.0	5,0	
		Filter	2003	26		362	36	257	255	251	関係	25%	366	36	253	361	360	359	1795	364	258	360	
		Probe	000	900	(Q	232	3/3		244	250	25%	7.34	25%	38 (	373	366	259	36	300	300	388	128	
ature, °F	Aeter	Outlet .	32	84	23	34	PH 94	44	45	4%	187	85	25	35	3	35	85	48	78	, 48	85	83	
Temperature,	Gas Meter	Inlet	86	25	89	89	89	96	88	02	90	2	80	90	4	89	8	8	47	89	96	0	
	Impinger	Outlet	59	50	50	49	50	52	49	15	12	57	33	49	48	5	49	43	177	49	7	511	
		Stack	391	399	400	396	395	382	390	391	198	76383	407	407	404	407	70h	410	413	412	<u>z</u>	413	
Gas Meter	ft <sup>3</sup>	150.407	153.63	756.95	81-096	763,31	362	11.691	772.38	775.36	778.43	781,176	74.47	16.786	741 47	795.05	798.293	45/108	404.76	808.17	311. 4(	814,303	
Orifice	Setting	$(\Delta H, \text{ in. } H_2O)$	2,0	5,0	2,1	00:	(.3	300	118	1.8	(7)	ゴニ	<u></u>	6.4	7	Le Le	į	9.1	7,	7.6	3.0	7	
Velocity	Head	$(\Delta p, \text{in. H}_2O)$	0.53	6.53	0.54	0.47	0,34	6.47	0,43	24,0	) H. a	0,33	0.50	65.0	0.53	0.59	0.4	15.0	05.0	0.50	0,47	0.33	
Time	(minutes)	1335 0	.3	8	4	_9	545 20	-5 (248 24	38	20	36	1309 UN	1310 44	85	20	20	1330 (10)	133661	69	73	78	30	
Traverse	Point	(Port-Pt)	05	4-	-3	-2	-1	-5	4-	-3	-2	-1	B -5	4-	-3	<sup>2</sup> -	-1	5- A <b>6</b>	4-	6	-2	<u>-</u> 1	Avg/Total

# Methods 1-5/26A Source Testing Field Data Sheet for Hydrogen Fluoride

Run 3

Source Testing Field Data Sheet for Hydrogen Fluoride	a Sheet for Hydro		Methods 1-5/26A	Run	اما	Page 0f	of
Client:Solvay Chemical	Meter ID 4	Date: 4/19/13	Impinger Weight	ight		Impinger	ger
Plant: Green River	Meter Y 0.997	Ambient Temp. °F: 92	Pretest (g) Post test (g) Total (g) Vol. (ml) Style* Contents	g) Total (g)	Vol. (ml)	Style* (	Contents
Test Location: Calciner	Meter $\Delta H @ 3 209$	Bar. Pressure in. Hg: 33.7	1762,2 992,2 230,0 ~ 100 MGS* 0.1N H2SO4	2300	001~	MGS*	0.1N H <sub>2</sub> SO <sub>4</sub>
Unit: CA 1 & 2 (aka CA- A & B)	Pitot ID p-4, 2	Static Press. in. H <sub>2</sub> O + & O, C, O	27881 869,6 815	81.5	2/100	GS*	~/CO GS* 0.1N H <sub>2</sub> SO <sub>4</sub>
Project No.: 1301A	Pitot Cp O, 94	Assumed % Moist	3 657.9 665.8 6.0	ço	- Statement	MGS*	
Meter Operator: J Russell	Probe Liner Glass	K Factor 4, 7	4963,6 976,9 13.3	_	MGS* Silica Gel	MGS*	Silica Gel
Assistant: D Klugar	Nozzle ID (5/6,55	Duct Dimensions: 144 inches	22	-			
Nozzle Calibration	Nozzle Size 6,304	Port Length: 3.5 inches 9.5	Filter teflon				
	Sample Time 30	Pitot Passes Leak Checks:			CO <sub>2</sub>	CO <sub>2</sub> CO <sub>2</sub> +O <sub>2</sub>	02
2nd Dia.	% CO2 & 9,0	Pretest Posttest	Diluent Analysis	1 <sup>st</sup> Orsat	9.0 22.4	22.4	13.4
3 <sup>rd</sup> Dia.	%0 <sub>2</sub> 13,4	1st Point all the way(In) Out	by CEMS	2 <sup>nd</sup> Orsat			

				,
	02	13,4		
	CO <sub>2</sub> CO <sub>2</sub> +O <sub>2</sub>	22.H		
	CO2	0,9		
		1 <sup>st</sup> Orsat	2 <sup>nd</sup> Orsat	
TANK TO THE TANK T		Diluent Analysis	$by$ CEMS $\Box$	
(11)	cks:	osttest 🗹	Out	

Leak Rates/Notes	Pre-test 7 in Hg 0,057cfm.	Post-test (O in Hg 0,000 cfm																				Ontimal Air Tasting	Opullial All Tesuing
Pump	Vacuum	(in. Hg)	7.5	15	2,5	7,0	20	7.0	8,0	800	8,5	2.50	2,5	7.0	7.0	2.0	ۆر	2.5	7.5	9,0	8,1	2.5	3
		Filter	359	365	363	5	360	264	361	260	260	259	2354	759	339	359	) 9E	363	360	360	250	36	
		Probe	365	328	Sec	399	786	496	370	263	359	356	949	303	397	383	326	359	386	372	364	355	
rature, °F	Gas Meter	Outlet .	25	34	86	251	25	85	31	25	366	85	84	12	38	33	36	35	85	88	24	35	
Temperature,		Inlet	68	200	50	00	90	386	90	00	-6	2	90	200	0	9	91	33	3	9	90	9	
	Impinger	Outlet	600	57	53	2	Sie	5.5	25	5	84	70	200	25	8.7	9	15	ro Co	2	53	55	57	
		Stack	411	711	514	200	413	TOF	403	804	403	403	397	394	394	8393	392	101	407	400	407	397	
Gas Meter	ft³	814.357	81.83	831, 22	334,67	927.90	430-684	334,03	537,57	841.33	844.93	348, 334	351.57	854.83	858.06	861.21	864,000	967.47	870,94	974.56	818.05	830,969	
Oritice	Setting	$(\Delta H, \text{in. } H_2O)$	から	2.1	2.2	5.	ナン	2.1	2.3	3.5	3.5	٥	0.6	0		1.8	1.5	2,2	ų,	ı.c	4.8	5	
Velocity	Head	(Δp, in. H <sub>2</sub> O)	0.53	0.49	0.53	0,45	0.34	0.50	6.54	0.59	000	0.47	0.417	0.45	0.415	0.44	0.35	0,53	0.53	30,0	0.17	0.35	
Time	(minutes)		.7	Ĉ∂	5	9	1728 20	1430 34	3.3	33	2	1450 40	1452 44	35	Š	22	1517 60	1514 64	63	6	76	1534 SO	
Iraverse	Point	(Port-Pt)	X0 -5	4-	-3	-2	-	C5	4-	-3	-2	-1	6 -5	4-	-3	-2	-1	A5	4-	-3	-2	-1	9 Avg/Total



#### **APPENDIX B**

#### DATA ENTRY/DATA REDUCTION SPREADSHEETS

#### Hydrogen Fluoride Sampling Data

Method 26A

Sum

4/19/13



#### Run 1

Meter ID:	4	% CO <sub>2</sub>	9.4
Meter Y:	0.992	% O <sub>2</sub>	13.0
e Meter ΔH@:	2.008	Total Sample Minutes:	80.0
Pitot ID:	P-4-2	Sample Minutes/Point:	4.0
Pitot Cp:	0.84	Barometric Pres., (in Hg):	23.70
Nozzle ID:	glass	Static Pressure (In. H <sub>2</sub> O):	-0.60
Nozzle Dia:	0.304	Dimensions (in)	144.0
		Area (ft <sup>2</sup> ):	113.097
		Filter:	Teflon
		Moisture	23.5%
	,	- All and a second a second and	
	Meter Y: e Meter ΔH@: Pitot ID: Pitot Cp: Nozzle ID:	Meter Y: 0.992 e Meter ΔH@: 2.008 Pitot ID: P-4-2 Pitot Cp: 0.84 Nozzle ID: glass	Meter Y: 0.992 % $O_2$ e Meter ΔH@: 2.008 Total Sample Minutes:  Pitot ID: P-4-2 Sample Minutes/Point:  Pitot Cp: 0.84 Barometric Pres., (in Hg):  Nozzle ID: glass Static Pressure (In. H <sub>2</sub> O):  Nozzle Dia: 0.304 Dimensions (in)  Area ( $ft^2$ ):  Filter:

306.5

Sar	npling	Time	Velocity	Orifice	Gas Meter	Ī	emperati	ıres	
Tra	averse	Minutes	Head, Δp	Setting	(ft³)		Gas	Meter	%
Port	<u>Point</u>	10:44	(in H2O)	(ΔH, in H2O)	688.765	Stack	Inlet	Outlet	<u>Iso</u>
D	5	10:48	0.51	2.0	691.83	409	80	79	89
	4	10:52	0.52	2.0	694.97	409	76	79	91
	3	10:56	0.51	2.0	698.11	411	75	79	92
	2	11:00	0.44	1.7	701.07	412	78	79	93
	1	11:04	0.35	1.4	703.846	410	79	79	97
	Re-Start	11:06			703.846				
С	5	11:10	0.50	2.0	706.87	401	81	79	88
	4	11:14	0.55	2.2	710.19	402	82	79	93
	3	11:18	0.60	2.3	713.59	401	85	81	90
	2	11:22	0.56	2.2	716.95	402	85	80	92
	1	11:26	0.46	1.8	720.024	400	87	80	93
	Re-Start	11:28			720.024				
В	5	11:32	0.47	1.8	723.09	393	86	83	91
	4	11:36	0.45	1.8	726.12	393	89	82	92
	3	11:40	0.45	1.8	729.18	392	89	83	93
	2	11:44	0.44	1.7	732.13	391	89	84	90
	1	11:48	0.37	1.4	734.84	391	90	85	90
	Re-Start	11:51			734.84				
A	5	11:55	0.53	2.0	738.05	403	89	84	90
	4	11:59	0.53	2.0	741.23	402	90	84	89
	3	12:03	0.57	2.2	744.59	401	89	84	91
	2	12:07	0.52	2.0	747.81	401	90	85	91
	1	12:11	0.27	1.0	750.20	395	89	84	93
Total/A	vg			1.87	61.435	401	SOL	VAY2	016_

## $\label{eq:continuous} \begin{tabular}{ll} Hydrogen Fluoride Sampling Data \\ Method 26A \end{tabular}$

316.6



6\_002654

4/19/13

Run 2

Sum

Solvay	Chemic	al		Meter ID:	4	% CO <sub>2</sub>	9.4
Source	e: CA - 1	& 2		Meter Y:	0.992	% O <sub>2</sub>	13.4
Test Location: Verical Stack (insi-				Meter ΔH@:	2.008	Total Sample Minutes:	80.0
OATS	Project	No. 1301.	A	Pitot ID:	P-4-2	Sample Minutes/Point:	4.0
Techni	cian: D. K	lassen, J. R	Russell	Pitot Cp:	0.84	Barometric Pres., (in Hg):	23.70
Moistu	re Data			Nozzle ID:	glass	Static Pressure (In. H <sub>2</sub> O):	-0.61
Imping	ei Pretest	Posttest	Total	Nozzle Dia:	0.304	Dimensions (in)	144.0
1	751.4	975.6	224.2			Area (ft²):	113.097
2	629.3	701.3	72.0			Filter:	Teflon
3	656.2	662.8	6.6			Moisture	23.5%
4	907.7	921.5	13.8				·

San	npling	Time	Velocity	Orifice	Gas Meter	T	emperatu	res	
Tra	verse	Minutes	Head, Δp	Setting	(ft <sup>3</sup> )		Gas	Meter	%
Port	Point	12:25	(in H2O)	(ΔH, in H2O)	750.407	Stack	Inlet	Outlet	<u>Iso</u>
A	5	12:29	0.53	2.0	753.63	391	86	85	90
	4	12:33	0.53	2.0	756.85	399	85	84	91
	3	12:37	0.54	2.1	760.18	400	89	85	93
	2	12:41	0.47	1.8	763.31	396	89	84	93
	1	12:45	0.34	1.3	765.962	395	89	84	93
	Re-Start	12:50			765.962				
В	5	12:54	0.47	2.0	769.16	382	90	84	95
	4	12:58	0.43	2.2	772.38	390	89	85	100
	3	13:02	0.42	1.8	775.36	391	90	84	94
	2	13:06	0.41	1.7	778.43	391	90	84	98
	1	13:10	0.33	1.4	781.176	383	91	85	97
	Re-Start	13:13			781.176				
С	5	13:17	0.50	2.1	784.47	402	89	85	96
	4	13:21	0.53	2.2	787.91	402	90	85	97
	3	13:25	0.58	2.4	791.47	404	92	86	96
	2	13:29	0.58	2.4	795.05	403	89	85	97
	1	13:33	0.41	1.7	798.09	402	91	85	97
	Re-Start	13:38			798.093				
D	5	13:42	0.51	2.1	801.420	410	86	84	96
	4	13:46	0.50	2.1	804.76	413	87	84	98
	3	13:50	0.51	2.1	808.12	412	89	84	97
	2	13:54	0.47	2.0	811.41	415	90	85	99
	1	13:58	0.33	1.4	814.203	412	90	85	100
Total/A	Total/Avg				63.796	400	SOL	<b>EVAY</b>	2016

#### Hydrogen Fluoride Sampling Data

#### Method 26A

4/19/13

#### Run 3



Solvay C	hemic	al		Meter ID:	4	% CO <sub>2</sub>	9.0
Source:	CA - 1	& 2		Meter Y:	0.992	% O <sub>2</sub>	13.4
Test Loc	ation:	Verical Sta	ack (inside	Meter ΔH@:	2.008	Total Sample Minutes:	80.0
OATS P	roject	No. 1301A		Pitot ID:	P-4-2	Sample Minutes/Point:	4.0
Technicia	n: D. K	lassen, J. Ru	ssell	Pitot Cp:	0.84	Barometric Pres., (in Hg):	23.70
Moisture 1	<u>Data</u>			Nozzle ID:	glass	Static Pressure (In. H <sub>2</sub> O):	-0.60
Impinger F	retest	Posttest	<u>Total</u>	Nozzle Dia:	0.304	Dimensions(in)	144.0
1	762.2	992.2	230.0			Area (ft <sup>2</sup> ):	113.097
2	788.1	869.6	81.5			Filter:	Teflon
3	659.8	665.8	6.0			Moisture	23.5%
4	963.6	976.9	13.3		,		
Sum 330.8							

San	npling	Time	Velocity	Orifice	Gas Meter	<u>T</u>	emperati	ıres	
Tra	verse	Minutes	Head, ∆p	Setting	(ft³)		Gas	Meter	%
<u>Port</u>	<u>Point</u>	14:08	(in H2O)	(ΔH, in H2O)	814.357	Stack	<u>Inlet</u>	Outlet	<u>Iso</u>
A	5	14:12	0.53	2.2	817.83	411	87	85	98
	4	14:16	0.49	2.1	821.22	411	89	84	100
	3	14:20	0.53	2.2	824.67	413	89	86	98
	2	14:24	0.45	1.9	827.900	413	90	85	99
	1	14:28	0.34	1.4	830.684	412	90	85	98
	Re-Start	14:30			830.684				
В	5	14:34	0.50	2.1	834.03	402	89	85	97
	4	14:38	0.54	2.3	837.57	403	90	84	99
	3	14:42	0.59	2.5	841.23	403	90	85	98
	2	14:46	0.60	2.5	844.93	403	91	86	98
	1	14:50	0.47	2.0	848.234	403	90	85	99
	Re-Start	14:52			848.234				
С	5	14:56	0.47	2.0	851.57	397	89	84	99
	4	15:00	0.45	1.9	854.83	394	90	85	99
	3	15:04	0.45	1.9	858.06	394	90	86	98
	2	15:08	0.44	1.8	861.21	392	91	85	96
	1	15:12	0.35	1.5	864.090	392	91	86	99
	Re-Start	15:14			864.090				
D	5	15:18	0.53	2.2	867.47	401	89	85	95
	4	15:22	0.53	2.2	870.94	402	91	85	97
	3	15:26	0.56	2.4	874.56	400	91	85	99
	2	15:30	0.52	2.2	878.05	402	90	84	99
	1	15:34	0.35	1.5	880.969	397	91	85	100
Total/A	vg			2.04	66.612	402	201	<sup>87</sup>	046

<del>SOLVAY2016\_</del>6\_002655



Source, CA - 1 & 2					
	Start Date/Time	4/19/13 10:44	4/19/13 12:25	4/19/13 14:08	
	Stop Date/Time	12:11	13:58	15:30	
Coal Consumption	<u>Units</u>	Run 1	Run 2	Run 3	
AQD #17 "A" Calciner	TPH	5.6	5.6	5.6	
AQD #17 "B" Calciner	TPH	5.6	5.6	5.6	
Combined Coal Consumption	TPH	11.2	11.2	11.2	
Test Parameters	<b>Units</b>	Run 1	Run 2	Run 3	Avg.
P <sub>bar</sub> (Barometric Pressure, absolute)	In. Hg	23.70	23.70	23.70	
Y (Dry Gas Meter Calibration Factor)	unitless	0.992	0.992	0.992	
C <sub>p</sub> (Pitot tube Coefficient)	unitless	0.84	0.84	0.84	
D <sub>n</sub> (Diameter of Nozzle)	Inches	0.304	0.304	0.304	
θ (Total Sampling Time of Test)	Minutes	80	80	80	
ΔΗ (Orifice Pressure Drop)	In. H2O	1.87	1.94	2.04	
V <sub>m</sub> (Dry Gas Sampled - as measured)	ft3 (dry)	61.435	63.796	66.612	
T <sub>m</sub> (Gas Meter Temperature, avg.)	Degr. F	83	87	87	
V <sub>lc</sub> (Condensate and silica gel)	ml or g	306.5	316.6	330.8	
Location/Process Parameters					
A <sub>s</sub> (Cross-sectional Area of Stack)	ft2	113.1	113.1	113.1	113.1
P <sub>g</sub> (Static Pressure of Stack Gas)	In. H2O	-0.60	-0.61	-0.60	-0.60
T <sub>s</sub> (Temperature of Stack Gas)	Deg. F	401	400	402	401
$\sqrt{\Delta p}$ (Sq. root of velocity head of gas)	√In. H2O	0.6902	0.6829	0.6940	0.6890
CO <sub>2</sub> (Carbon Dioxide)	%	9.4	9.4	9.0	9.3
O <sub>2</sub> (Oxygen)	%	13.0	13.4	13.4	13.3
Calculations					
V <sub>mstd</sub> (Gas Sampled, standard (std) cond.)	$\mathrm{ft}^3$	47.17	48.68	50.78	48.88
V <sub>wstd</sub> (Water Vapor in Gas Sampled, std)	ft <sup>3</sup>	14.45	14.93	15.60	14.99
B <sub>ws</sub> (Water Vapor in Gas, by Vol.)	%	23.45	23.47	23.50	23.47
M <sub>d</sub> (Molecular Weight of Dry Stack Gas)	lb/lb-mole	30.02	30.04	29.98	30.01
M <sub>s</sub> (Molecular Weight of Wet Stack Gas)	lb/lb-mole	27.20	27.21	27.16	27.19
P <sub>s</sub> (Pressure of Stack Gas, Absolute)	In. Hg	23.66	23.66	23.66	23.66
Iso (Percent of Isokinetic Sampling)	%	91.7	95.6	98.2	95.2
Flow Results					
V <sub>s</sub> (Average Stack Gas Velocity)	ft/m (fpm)	3,440	3,400	3,464	3,435
Q <sub>a</sub> (Actual Volumetric Flow Rate)	ft <sup>3</sup> /m (cfm)	389,040	384,540	391,750	388,443
Q <sub>std</sub> (Dry Volumetric Flow Rate, std.)	ft <sup>3</sup> /m (dscfm)	144,340	142,850	145,040	144,077
HF Results					
Mass of HF Collected	mg/sample	< 0.123	< 0.126	< 0.128	
HF Concentrations in gas - std.	mg/m³ gas	< 0.092	< 0.094	< 0.096	
HF Concentrations in gas - std.	gr/dscf	< 0.00004	< 0.00004	< 0.00004	30
HF Concentrations in gas	ppm	< 0.111	< 0.113	< 0.115	< 0.113
HF Emission Rate (Coal Fd = 9780)	lb/mmBtu	<1.49E-04	<1.56E-04	<1.51E-04	<1.52E-04
HF Emission Rate	lb/hr	< 0.050	< 0.049	<0.048	<0.049
HF Emission Rate	lb/ton of coal	< 0.0044	< 0.0044	<0.0043	<0.0044



# APPENDIX C EQUIPMENT CALIBRATIONS

#### Calibrations for Meter Box 4

F. II T. ( )							
Full Test Meter Calibration - Critical Orifice							
Technician: Gene Wintermote							
1/3/2013	4.0	45					
Run No.	<u>1A</u>	<u>1B</u>	<u>2A</u>	<u>2B</u>	<u>3A</u>	<u>3B</u>	<u>Average</u>
Barometric Pressure, Pb	24.90	24.90	24.90	24.90	24.90	24.90	
Calibration Orifice Coef. (K)	0.6810	0.6810	0.5560	0.5560	0.4240	0.4240	
Final Meter Reading, ft <sup>3</sup>	274.742	280.851	287.225	293.611	299.035	304.556	
Initial Meter Reading, ft <sup>3</sup>	268.597	274.742	280.854	287.225	293.611	299.035	
Total Metered Volume, ft <sup>3</sup>	6.145	6.109	6.371	6.386	5.424	5.521	
Initial Inlet Meter Temp, °F	48	50	50	51	51	52	
Final Inlet Meter Temp, °F	50	50	50	51	51	53	
Initial Outlet Meter Temp, °F	48	49	50	50	51	52	
Final Outlet Meter Temp, °F	49	50	50	51	51	52	
Average Meter Temp, °R	509	510	510	511	511	512	
Time: Minutes of Run Time	7	7	9	9	10	10	
Seconds of Run Time	0.00	0.00	0.00	0.00	0.00	0.00	
Initial Orifice pressure drop, ΔH	2.30	2.30	1.50	1.50	0.92	0.92	
Final Orifice pressure drop, ΔH	2.30	2.30	1.50	1.50	0.92	0.92	
Avg. Orifice pressure drop, ∆H	2.30	2.30	1.50	1.50	0.92	0.92	
							w.
Ambient (Orifice) Temp., °F	52	51	51	52	52	53	
Vacuum Setting, "Hg	12.0	12.0	14.0	14.0	15.5	15.5	
V <sub>cr std</sub>	5.246	5.251	5.512	5.507	4.666	4.661	
Std, Volume Metered, Q <sub>std</sub> , ft <sup>3</sup>	5.341	5.300	5.511	5.516	4.675	4.747	
Calibration Factor (Y)	0.982	0.991	1.000	0.998	0.998	0.982	0.992
Tolerance within allowable limits	Pass	Pass	Pass	Pass	Pass	Pass	
Orifice Cal. Factor, ∆H @	2.017	2.009	1.955	1.956	2.055	2.054	2.008
Tolerance within allowable limits	Pass	Pass	Pass	Pass	Pass	Pass	

Thermocouple Calibrations							
Technician: G	. Winterm	ote					
Date of Calibration: 8	/22/2012						
		Impinger	Impinger Dry Gas Meter				
	Stack	Outlet	Inlet	Outlet	Auxilary	Probe	Filter
lce					-		
Temp. of Reference, °F	32	32	32	32	32	32	32
Thermocouple Reading, °F	32	30	32	33	30	30	32
% Difference, based on °R	0.00	0.41	0.00	-0.20	0.41	0.41	0.00
Boiling Water							
Temp. of Reference, °F	212	212	212	212	212	212	212
Thermocouple Reading, °F	211	212	213	212	212	209	213
% Difference, based on °R	0.15	0.00	-0.15	0.00	0.00	0.45	-0.15
<u>Oil</u>							
Temp. of Reference, °F	449	449	449	449	449	449	449
Thermocouple Reading, °F	450	449	450	448	448	447	450
% Difference, based on °R	-0.11	0.00	-0.11	0.11	0.11	0.22	-0.11

#### Type S Pitot Tube Inspection Data



Date:	10-Apr-13 Pito	ot Number:	P-4-2		
Pitot Tube Assembly Level?	yes	X	no		no position of the control of the co
Pitot Tube Assembly Damaged? If yes explain below.	yes		no	X	realities.
$\alpha_1$	0.9 (<10	°)	$\alpha_2$	0.5	(<10°)
$\beta_1$	2.1 (<5°,	)	$\beta_2$	2.8	(<5°)
$\gamma =$	0.5		$\theta = $	1.6	0
A =	0.735 incl	nes			
$Z = A SINE \gamma =$	0.0064 incl	nes	Where $Z$ is $< 0.32$ cm (	'<1/8 in)	
$W = A SINE \theta =$	0.0205 incl	nes	Where $W$ is $< 0.08$ cm	(<1/32 in)	
Pa =	0.376 incl	nes	Pb =	0.370	inches
P =	(Pa + Pb) / 2 = 0.37	'3 inches			
Dt =	0.25 incl	hes	P/Dt = 1	492 inches	Where $1.05 \le P/Dt \le$
Comments:	Meets geometric ca	libration re	quirements.		
	Cp = 0.84				
	48 inch	effective pr	obe		
Additional Calibra	ation Required?	yes		no	

Calibrated by: Justin Russell



# APPENDIX D EXAMPLE EQUATIONS



#### Abbreviations and Nomenclature for Emissions Calculations

A	Cross 2224 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2		~
$A_n$	Cross sectional area of nozzle, (ft <sup>2</sup> )	$P_{std}$	Standard absolute barometric pressure,
$A_s$	Cross sectional stack Area, (ft <sup>2</sup> )		(29.92 in. Hg)
$B_{ws}$	Proportion of water vapor, by volume, in the gas stream	P <sub>static</sub>	Difference between stack gas pressure and
C	T	D	P <sub>bar</sub> (in. H <sub>2</sub> O)
$C_{gas}$	Drift Corrected pollutant or diluent	$P_s$	Absolute stack gas pressure, (in. Hg)
0	concentration on a dry basis	$\Delta p$	Velocity head reading at traverse point,
$C_{run}$	Average pollutant/diluent concentration		(in. H <sub>2</sub> O)
0	reported by analyzer for run, dry basis	ppm	Parts per million
$C_{m}$	Average of initial and final system	$Q_a$	Actual volumetric flow rate, at stack gas
	calibration bias check responses for the		conditions (acfm)
	upscale calibration gas, ppm	$Q_{std}$	Volumetric flow rate at dry standard
$C_0$	Average of initial and final system		conditions (dscfm)
	calibration bias check responses for the	$T_{m}$	Absolute temp. of stack gas at meter (°R,
	zero gas, ppm	- 111	avg. of meter inlet & outlet temps)
$C_{ma}$	Actual concentration of the upscale	$T_{S}$	Absolute temperature of stack gas (°R)
	calibration gas, ppm		
CE	Calibration Error, Difference between	θ	Total Sampling Time, minutes
	analyzer reading and calibration gas	$v_{\rm s}$	Velocity of stack gas (ft/sec)
	injected directly into analyzer	$V_{lc}$	Volume of liquid condensed in impingers
CF	Cubic Feet, ft <sup>3</sup>		and silica gel (grams or ml)
CFM	Cubic Feet per Minute, ft <sup>3</sup> /min.	Vm	Dry gas volume measured by dry gas
$C_p$	Pitot Tube Coefficient, dimensionless		meter (cubic feet)
$C_{PM}$	Particulate matter concentration, corrected	$Vm_{std}$	Dry gas volume measured by dry gas
CPIVI	to standard condition, lb/dscf		meter, standard conditions (dscf)
$D_n$	Diameter of nozzle, inches (in)	$Vw_{std}$	Volume of water vapor sampled, at
$D_{s}$	Diameter of stack, ft		standard conditions (cubic feet)
		W	Width of duct
$E_{PM}$	Particulate matter emission rate, lb/hr	32	Molecular weight of O <sub>2</sub> , (lb/ <sub>lb-mole</sub> )
Egas	Gaseous Pollutant emission rate, lb/hr	44	Molecular weight of CO <sub>2</sub> , (lb/ <sub>lb-mole</sub> )
ΔΗ	Pressure Differential across meter orifice	28	Molecular weight of N <sub>2</sub> (lb/lb-mole)
in. Hg	Pressure, measured as inches of Mercury	64.06	Molecular weight of SO <sub>2</sub> , (lb/ <sub>lb-mole</sub> )
%I	Percent Isokinetic	46.01	Molecular weight of NOx, (lb/lb-mole)
$K_p$	Velocity pressure coefficient, 85.49 (ft/sec)	17.64	Conversion factor, (°R/in. Hg)
N ANY	[(lb/lb-mole) (in. Hg/°R) (1/in. H <sub>2</sub> O)] <sup>1/2</sup>	13.6	Conversion factor, (in.H <sub>2</sub> O/in. Hg)
$MW_s$	Molecular Wt of gas sample, (lb/lb-mole)	460	Conversion factor, (°F to °R)
$MW_d$	Molecular wt of dry flue gas, (lb/lb-mole)		Conversion factor for isokinetic calc.
$MW_w$	Molecular weight of water, (18 lb/lb-mole)	$10^6$	Conversion Factor, parts per million
$m_{PM}$	Particulate matter mass, (grams)	385.3	
$\%O_2$	Percent of oxygen in flue gas by volume,		Conversion factor, (dscf/ <sub>lb-mole</sub> )
	dry basis	15.43	Conversion factor, (grains/gram)
$%CO_2$	Percent of carbon dioxide in flue gas by	7000	Conversion factor, (grains/pound)
	volume, dry basis		
$%N_2$	Percent of nitrogen in flue gas by volume,		
	dry basis		
$P_{bar}$	Absolute barometric pressure at sampling		
	location, (in. Hg)		



#### Sample Calculations - Run 1

Raw data is entered into spreadsheets to calculate results electronically. Calculations shown below use the spreadsheet generated results. Calculations with a calculator may not exactly match the results shown because of significant figures.

1. Circular Stack Area

$$A_{s} = \frac{\pi}{4} (D_{s})^{2} = \frac{\pi}{4} \left(\frac{144}{12}\right)^{2} = 113.10 ft^{2}$$

2. Absolute stack gas pressure

$$P_{s} = \left(P_{bar} + \frac{P_{static}}{13.6}\right) = \left((23.70) + \frac{-0.60}{13.6}\right) = 23.66in.Hg$$

3. Volume of dry gas sampled at standard conditions (29.92"Hg, 528°R)

$$Vm_{std} = \left(\frac{17.64(V_m)(P_b + \frac{\Delta H}{13.6})}{T_m}\right)(Y_d) = \left(\frac{17.64(61.435)(23.70 + \frac{1.87}{13.6})}{460 + 83}\right)(0.992) = 47.17 dscf$$

4. Volume of water vapor collected at standard conditions

$$Vw_{std} = 0.04715V_{lc}$$
 = 0.04715(306.5) = 14.45scf

5. Proportion, by volume, of water vapor to dry gas, percent

$$B_{ws} = \frac{Vw_{std}}{(Vw_{std} + Vm_{std})} = \frac{14.45}{(14.45 + 47.17)} = 23.45\%$$

6. Dry molecular weight of stack gas

$$M_{d} = \left(\frac{44}{100}\right)\% CO_{2} + \left(\frac{32}{100}\right)\% O_{2} + \left(\frac{28}{100}\right)\% N_{2} = 0.44(9.4) + 0.32(13.0) + 0.28(77.6) = 30.02 \frac{lb}{lb-mole}$$

7. Actual molecular weight of stack gas

$$M_s = (1 - B_{ws})(M_d) + MW_w(B_{ws})$$
 =  $(1 - 0.2345)(30.02) + 18(0.2345)$  =  $27.20 \frac{lb}{lb-mole}$ 



8. Velocity of stack gas

$$V_s = K_p(C_p)\sqrt{\Delta p} 60 \sqrt{\frac{T_s}{(P_s)(M_s)}} \\ = 85.49(0.84)(0.6902)60 \sqrt{\frac{(401 + 460)}{(23.66)(27.20)}} \\ = 3,440 \frac{feet}{min\ ute}$$

9. Actual stack gas volumetric flow rate

$$Q_a = v_s(A_s)$$
 = 3440(113.10) = 389,040  $\frac{G}{\min ule}$ 

10. Stack gas volumetric flow rate at standard conditions

$$Q_{std} = \frac{17.64Q_{a}P_{s}\left(1 - \frac{B_{ws}}{100}\right)}{(T_{s})} = \frac{17.64(389,040)(23.66)\left(1 - \frac{23.45}{100}\right)}{(401 + 460)} = 144,340 \frac{dscf}{min ute}$$

11. Sampling Nozzle Area

$$A_{n} = \frac{\pi}{4} (D_{n})^{2} = \frac{\pi}{4} \left(\frac{0.304}{12}\right)^{2} = 0.00050 ft^{2}$$

12. Percent of isokinetic sampling

$$\%I = \frac{0.09450 (T_s) V m_{std}}{P_s v_s A_n \theta (1 - B_{ws})} = \frac{0.09450 (401 + 460) (47.17)}{23.66 \left(\frac{3440}{60}\right) (0.00050) 80 (1 - 0.2345)} = 91.7\%$$

13. Hydrogen Fluoride Concentration in gas (mg/m<sup>3</sup>)

$$C_{HF} = \frac{(M_{HF})(35.316)}{(V_{mstd})}$$

$$C_{HF} = \frac{(0.123)(35.316)}{(47.17)}$$

$$C_{HF} = 0.092 \, mg \, / m^3$$

Where:

m<sub>HF</sub> hydrogen fluoride concentration (mg)

V<sub>mstd</sub> Dry gas volume measured by dry gas meter, standard conditions (dscf)

C<sub>HF</sub> Concentration of hydrogen fluoride



14. Hydrogen Fluoride Concentration (gr/dscf)

$$C_{gr/dscf} = \frac{(m_{HF})(0.01543)}{(V_{mstd})}$$

$$C_{gr/dscf} = \frac{(0.123)(0.01543)}{(47.17)}$$

$$C_{gr/dscf} = 0.000040 \, gr/dscf$$

Where:

 $m_{HF}$ 

hydrogen fluoride concentration (mg)

 $V_{mstd}$ 

volume of gas sample, corrected to standard conditions (scf)

0.01543

conversion factor

15. Hydrogen Fluoride Concentration (ppm)

$$C_{ppm} = \frac{\left(C_{gr/dscf}\right)(24.04)}{MW_{HF}}$$

$$C_{ppm} = \frac{\left(0.092\right)(24.04)}{20.00634}$$

$$C_{ppm} = 0.111ppm$$

Where:

 $C_{ppm}$ 

hydrogen fluoride concentration (ppm)

C<sub>gr/dscf</sub>

hydrogen fluoride concentration (gr/dscf)

 $MW_{HF}$ 

molecular weight of hydrogen fluoride (lb/lb-mole)



#### 16. Hydrogen Fluoride Emission (lb/hr)

$$E_{lb/hr} = (0.01543) \left(\frac{M_{HF}}{V_{mstd}}\right) \left(\frac{60}{7000}\right) (Q_{std})$$

$$E_{lb/hr} = (0.01543) \left(\frac{60}{7000}\right) \left(\frac{0.123}{47.17}\right) (144340)$$

$$E_{lb/hr} = 0.050 lb/hr$$

#### Where:

E<sub>lb/hr</sub>

hydrogen fluoride emission (lb/hr)

 $M_{\text{HF}}$ 

hydrogen fluoride mass

 $V_{mstd}$ 

Dry gas volume measured by dry gas meter, standard conditions (dscf)

Q<sub>std</sub>

volumetric flow rate of gas stream at standard conditions, dry basis (dscfm)

#### 17. Hydrogen Fluoride Emission (lb/mmBtu, F<sub>d</sub> Factor)

$$E_{lb/mmBtu} = \frac{(HF_{gr/dscf})(9780)(20.9)}{(7000)(20.9 - \%O_2)}$$

$$E_{lb/mmBtu} = \frac{(0.00004)(9780)(20.9)}{(7000)(20.9 - 13.0)}$$

 $E_{lb/mmBtu} = 0.000149 lb/mmBtu$ 

#### Where:

 $E_{lb/mmBtu}$ 

hydrogen fluoride emission (lb/mmBtu)

HF<sub>gr/dscf</sub>

hydrogen fluoride concentration in gas (gr/dscf)

20.9

oxygen concentration in ambient air (%)

 $%O_2$ 

average concentration of oxygen for the test run (%)



# APPENDIX E LABORATORY DATA

# **Optimal Air Testing Services**

9971 W. Landmark Lane Casper, WY 82604

Solvay Chemical
Client Project #1301A

Analytical Report (0413-135)

EPA Method 26A

Hydrogen fluoride



#### **Enthalpy Analytical, Inc.**

Phone: (919) 850 - 4392 / Fax: (919) 850 - 9012 / www.enthalpy.com 800-1 Capitola Drive Durham, NC 27713-4385

SOLVAY2016\_6\_00266

I certify that to the best of my knowledge all analytical data presented in this report:

- Have been checked for completeness
- Are accurate, error-free, and legible
- Have been conducted in accordance with approved protocol, and that all deviations and analytical problems are summarized in the appropriate narrative(s)

This analytical report was prepared in Portable Document Format (.PDF) and contains 61 pages.

QA Review Performed by: Michael Steven Schapira

Report Issued: 4/29/13



# **Summary of Results**



Company	OATS
Analyst	EDE
Parameters	EPA Method 26A

Client # 1301A Job # 0413-135 # Samples 3, 2 Blanks

Compound

Sample ID / Catch Weight (ug)

SC-M26A-R1-H2SO4

SC-M26A-R2-H2SO4

SC-M26A-R3-H2SO4

Hydrogen fluoride

Hydrogen fluoride

123 ND

126 ND

128 ND

RB-1-H2SO4

45.1 ND

**RB-2-Water** 45.5 ND

SOLVAY2016\_6\_002670

# Results



OATS EDE EPA Method 26A Client # 1301A Job # 0413-135 # Samples 3, 2 Blanks

Lower Curve Limit 0.200 (ug/mL) Upper Curve Limit 10.0 (ug/mL)

#### e as fluoride

											Or Control of the Control			
)	Lab ID # 2	Analysis Method	Ret Time (min)	Ret Time (min)	% Diff Ret	Conc #1 (ug/mL)	Conc # 2 (ug/mL)	% Diff Conc	Avg Conc (ug/mL)	DF	Vol (mL)	Conv. Factor	Catch Weight (ug)	Qual
1.D	051-2002.D	HPLC72PG111.M	NA	NA	NA	0.0200	0.0200	0.0	0.0200	10	586	1.053	123	ND
1.D	054-2302.D	HPLC72PG111.M	NA	NA	NA	0.0200	0.0200	0.0	0.0200	10	600	1.053	126	ND
11.D	055-2402.D	HPLC72PG111.M	NA	NA	NA	0.0200	0.0200	0.0	0.0200	10	610	1.053	128	ND
1.D	056-2502.D	HPLC72PG111.M	NA	NA	NA	0.0200	0.0200	0.0	0.0200	10	214	1.053	45.1	ND
1.D	057-2602.D	HPLC72PG111.M	NA	NA	NA	0.0200	0.0200	0.0	0.0200	10	216	1.053	45.5	ND
1.D	007-0902.D	HPLC72PG111.M	3.04	3.05	0.1	5.01	5.05	0.3	5.03	1	10.0	1.053	53.0	
										Sp	ike Am	ount (ug)	52.7	
										Sp	ike Rec	overy (%)	101%	
1.D	008-1002.D	HPLC72PG111.M	2.97	2.97	0.0	5.12	5.13	0.1	5.12	1	10.0	1.053	54.0	
										Sp	ike Am	ount (ug)	52.7	
										Sp	ike Rec	overy (%)	102%	

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4/29/2013

ATS				
DE				
EPA Method 26A				

Client #	1301A
Job#	
# Samples	3, 2 Blanks

Lower Curve Limit 0.200 (ug/mL) Upper Curve Limit 10.0 (ug/mL)

as fluoride

	Lab ID # 2	Analysis Method	Ret Time (min)	Ret Time (min)	% Diff Ret	Conc # 1 (ug/mL)	Conc # 2 (ug/mL)	% Diff Conc	Avg Conc (ug/mL)	DF	Vol (mL)	Conv. Factor	Catch Weight (ug)	Qual
D	009-1102.D	HPLC72PG111.M	NA	NA	NA	0.0200	0.0200	0.0	0.0200	1	1.00	1.053	0.0211	ND
1.D	052-2102.D	HPLC72PG111.M	3.05	3.05	0.2	3.06	3.10	0.6	3.08	1	10.0	1.053	32.4	
										Sp	ike Am	ount (ug)	31.6	
										Nat	ive Am	ount (ug)	0.00	
										Sp	ike Reco	overy (%)	103%	
D	053-2202.D	HPLC72PG111.M	3.05	3.05	0.0	3.10	3.10	0.0	3.10	1	10.0	1.053	32.6	
Spike Amount (ug)							31.6							
Native Amount (ug)							0.00							
Spike Recovery (%)							103%							

EA Job # 0413-135 Page 7 of 61

4/29/2013

# **Narrative Summary**



#### **Enthalpy Analytical Narrative Summary**

Company	Optimal Air Testing Services
Analyst	EDE
Parameters	EPA Method 26A

Client #	1301A	
Job#	0413-135	
# Samples	3, 2 Blanks	

#### Custody

Chester Burnett received the samples on 4/24/13 after being relinquished by Optimal Air Testing Services. The samples were received at 14.6°C and in good condition. Prior to, during, and after analysis, the samples were kept under lock with access only to authorized personnel by Enthalpy Analytical, Inc.

#### **Analysis**

The samples were analyzed for fluoride using the analytical procedures in EPA Method 26A, Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources Isokinetic Method (40 CFR Part 60, Appendix A).

The samples were analyzed following the procedures in Section 11.0, Analytical Procedures. All samples and standards are prepared, stored, and analyzed using high-density polyethylene containers.

The Metrohm 861 Compact IC ("Smithers" S/N 1861002007189) was equipped with a Metrohm 861 Conductivity Detector and a Metrosep A Supp 5 - 110/4.0mm (S/N # 7908297) column.

#### Calibration

The calibration curve is located in the back of this report and referenced in the Analysis Method column on the Detailed Results page.

For each calibration curve used, the first page of the curve contains all method specific parameters (i.e., curve type, origin, weight, etc.) used to quantify the samples. The calibration curve section also includes a table with the Retention Time (RetTime), Level (Lvl), Amount (corresponding units), Area, Response Factor (Amt/Area) and the analyte Name. The calibration table is used to identify (by retention time) and quantify each target compound.

#### Chromatographic Conditions

The acquisition method Metrohm.M is included in the Calibration Curve Chromatograms section of this report.

#### **OC** Notes

The samples were analyzed within the holding time specified by the method.

The analyses of the laboratory reagent blanks or client blanks contained no fluoride at concentrations greater than the detection limit.



### Enthalpy Analytical Narrative Summary (continued)

(	QC	N	ot	es
(c	on	tin	ue	ed)

Duplicate matrix spikes were prepared using aliquots of sample *SC-M26A-R1-H2SO4* and both exhibited spike recovery values of 103%.

Laboratory Control Samples (LCS) were analyzed along with the samples. The recovery values were 101% and 102%.

#### Reporting Notes

The sulfuric acid matrix samples were analyzed for fluoride but are reported as hydrogen fluoride. The results were converted using a conversion factor of 1.028 to account for the additional hydrogen mass.

The results presented in this report are representative of the samples as provided to the laboratory.



#### **General Reporting Notes**

The following are general reporting notes that are applicable to all Enthalpy Analytical, Inc. data reports, unless specifically noted otherwise.

- Any analysis which referrs to the method as "*Type*" represents a planned deviation from the reference method. For instance a Hydrogen Sulfide assay from a Tedlar bag would be labeled as "EPA Method 16-Type" because Tedlar bags are not mentioned as one of the collection options in EPA Method 16.
- The acronym *MDL* represents the Minimum Detection Limit. Below this value the laboratory cannot determine the presence of the analyte of interest reliably.
- The acronym *LOQ* represents the Limit of Quantification. Below this value the laboratory cannot quantitate the analyte of interest within the criteria of the method.
- The acronym ND following a value indicates a non-detect or analytical result below the MDL.
- The letter *J* in the Qualifier or Flag column in the results indicates that the value is between the MDL and the LOQ. The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.
- The letter *E* in the Qualifier or Flag column indicates an analytical result exceeding 100% of the highest calibration point. The associated value should be considered as an estimate.
- The acronym *DF* represents Dilution Factor. This number represents dilution of the sample during the preparation and/or analysis process. The analytical result taken from a laboratory instrument is multiplied by the DF to determine the final undiluted sample results.
- The addition of MS to the Sample ID represents a Matrix Spike. An aliquot of an actual sample is spiked with a known amount of analyte so that a percent recovery value can be determined. The MS analysis indicates what effect the sample matrix may have on the target analyte, i.e. whether or not anything in the sample matrix interferes with the analysis of the analyte(s).
- The addition of *MSD* to the Sample ID represents a Matrix Spike Duplicate. Prepared in the same manner as an MS, the use of duplicate matrix spikes allows further confirmation of laboratory quality by showing the consistency of results gained by performing the same steps multiple times.
- The addition of *LD* to the Sample ID represents a Laboratory Duplicate. The analyst prepares an additional aliquot of sample for testing and the results of the duplicate analysis are compared to the initial result. The result should have a difference value of within 10% of the initial result (if the results of the original analysis are greater than the LOQ).
- The addition of *AD* to the Sample ID represents an Alternate Dilution. The analyst prepares an additional aliquot at a different dilution factor (usually double the initial factor). This analysis helps confirm that no additional compound is present and coeluting or sharing absorbance with the analyte of interest, as they would have a different response/absorbance than the analyte of interest.
- The Sample ID *LCS* represents a Laboratory Control Sample. Clean matrix, similar to the client sample matrix, prepared and analyzed by the laboratory using the same reagents, spiking standards and procedures used for the client samples. The LCS is used to assess the control of the laboratory's analytical system. Whenever spikes are prepared for our client projects, two spikes are retained as LCSs. The LCSs are labeled with the associated project number and kept in-house at the appropriate temperature conditions. When the project samples are received for analysis, the LCSs are analyzed to confirm that the analyte could be recovered from the media, separate from the samples which were used on the project and which may have been affected by source matrix, sample collection and/or sample transport.



#### **General Reporting Notes**

(continued)

- **Significant Figures**: Where the reported value is much greater than unity (1.00) in the units expressed, the number is rounded to a whole number of units, rather than to 3 significant figures. For example, a value of 10,456.45 ug catch is rounded to 10,456 ug. There are five significant digits displayed, but no confidence should be placed on more than two significant digits.
- Manual Integration: The data systems used for processing will flag manually integrated peaks with an "M". There are several reasons a peak may be manually integrated. These reasons will be identified by the following two letter designations. The peak was not integrated by the software "NI", the peak was integrated incorrectly by the software "II" or the wrong peak was integrated by the software "WP". These codes will accompany the analyst's manual integration stamp placed next to the compound name on the chromatogram.



# **Sample Custody**



					Analysis / Pres	servative / PH	 
307) 237	oming 82604 -0961						Comments
4 21	7/3 Date	Contents	Containers	HF		Volume	
KHE							
-1	4/19/2013	0.1 N H2SO4	2	X		X	
-1	4/19/2013	0.1 N H2SO4	2	X		X	
-1	4/19/2013	0.1 N H2SO4	2	X		X	
	4/19/2013	0.1 N H2SO4	1	X			
	4/19/2013	Water	1	X			
					-		

ndix A Method 26A

e call to discuss quick turnaround)

ear minimum detection limits

		Receive		Reling	uish	
		Date	Time	Date	Time	Affiliation
Dan Klassen	DK			4/22/2013	10:00	OATS
Justin Russell	JR	4/22/2013	11:00			
Chesta Bunost		4-24-13	10:35A			

Temp=14.6% Ray Gun FF1

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APPENDIX F
PROCESS DATA

#### AQD #17 "A" Calciner

(CA-1/EP-1)

DATE: 4/19/13

#### **OPERATIONAL DATA**

	Run #1	Run #2	Run #3	<b>Average</b>
Start Time	10:44	12:25	14:08	
Stop Time	12:11	13:58	15:30	
Production Rate TPH	104	105	108	105.7
Opacity	0.4	0.3	0.2	0.3
Coal Consumption TPH	5.6	5.6	5.6	5.6

#### PRECIPITATOR DATA

	Run #1	Run #2	Run #3
FIELD 1			
SECONDARY VOLTAGE kV	0	0	0
SECONDARY CURRENT mA	0	0	0
FIELD 2			
SECONDARY VOLTAGE kV	34	35	34
SECONDARY CURRENT mA	161	154	154
FIELD 3			
SECONDARY VOLTAGE kV	31	32	21
SECONDARY CURRENT mA	1384	1389	1388
FIELD 4			
SECONDARY VOLTAGE kV	30	30	30
SECONDARY CURRENT mA	1352	1355	1357
FIELD 5			
SECONDARY VOLTAGE kV	29	30	30
SECONDARY CURRENT mA	1311	1312	1315
FIELD 6			
SECONDARY VOLTAGE kV	0	0	0
SECONDARY CURRENT mA	0	0	0

# AQD #17 "B" Calciner (CA-2/EP-2)

DATE: 4/19/13
OPERATIONAL DATA

	Run #1	Run #2	Run #3	Average
Start Time	10:44	12:25	14:08	
Stop Time	12:11	13:58	15:30	
Production Rate TPH	98	97	106	100.3
Opacity	0.4	0.3	0.2	0.3
Coal Consumption TPH	5.6	5.6	5.6	5.6

#### PRECIPITATOR DATA

	Run #1	Run #2	Run #3
FIELD 1			
SECONDARY VOLTAGE kV	29	28	29
SECONDARY CURRENT mA	174	165	166
FIELD 2			
SECONDARY VOLTAGE kV	32	32	32
SECONDARY CURRENT mA	1377	1379	1376
FIELD 3			
SECONDARY VOLTAGE kV	32	32	32
SECONDARY CURRENT mA	1402	1401	1379
FIELD 4			
SECONDARY VOLTAGE kV	26	26	25
SECONDARY CURRENT mA	395	404	404
FIELD 5			
SECONDARY VOLTAGE kV	30	30	30
SECONDARY CURRENT mA	1399	1400	1400
FIELD 6			
SECONDARY VOLTAGE kV	0	0	0
SECONDARY CURRENT mA	0	0	0